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PCT/US01/01973

INVERT WELL SERVICE FLUID AND METHOD

RELATED APPLICATION

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WO 01/59256

This application claims priority to U.S. Provisional Patent Application No. 60/181,439, filed February 10, 2000, and to U.S. Patent Application No. 09/641,635, filed August 17, 2000.

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BACKGROUND OF THE INVENTION

1. Field of the Invention

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This invention generally relates to compositions and methods or processes particularly suited for gravel packing and workover of wells drilled in subterranean hydrocarbon-bearing formations, including without limitation horizontal and directional wells. This invention also generally relates to compositions employed as displacement fluids, packer fluids, and fluids for drilling, especially drilling through pay zones of subterranean hydrocarbon-bearing formations.

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2. Description of Relevant Art

degrees Fahrenheit) holes.

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As used herein, the terms "well service fluid" or "wellbore fluid," unless indicated otherwise, shall be understood to mean a fluid used for sand control, gravel packing, and workover operations, as well as a fluid employed as a displacement fluid, a packer fluid, or a fluid for drilling and especially drilling through a pay zone. The term "solids-free" as applied to the basic well service fluid shall be understood to mean that no solid materials (e.g., weighting agents or commercial particulates) are present in the wellbore fluid (except that the term is not intended to exclude the presence of drill cuttings in the fluid in the well). The term "horizontal" with respect to a wellbore or to drilling shall be understood to mean at an angle or incline other than 90 degrees from the wellbore surface as the surface is viewed as a plane at ground level. The term "brine-sensitive formation" shall be understood to mean a formation sensitive to brines or that is best suited for oil-based drilling and well service

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fluids, such as a formation having swelling or sloughing shales, salt, gypsum, anhydrite or other evaporite formation, a hydrogen-sulfide containing formation and hot (greater than 300

A drilling fluid or mud is a specially designed fluid that is circulated through a wellbore as the wellbore is being drilled to facilitate the drilling operation. The various

functions of a drilling fluid include removing drill cuttings from the wellbore, cooling and lubricating the drill bit, aiding in support of the drill pipe and drill bit, and providing a hydrostatic head to maintain the integrity of the wellbore walls and prevent well blowouts. Specific drilling fluid systems are selected to optimize a drilling operation in accordance with the characteristics of a particular geological formation.

A drilling fluid comprising liquid, usually water and/or oil or synthetic oil or other synthetic material or synthetic fluid ("synthetic"), with solids in suspension is typically called a drilling fluid or mud. A non-aqueous based drilling fluid typically contains oil or a synthetic as the continuous phase and may also contain water which is dispersed in the continuous phase by emulsification so that there is no distinct layer of water in the fluid. Oil or synthetic-based muds are normally used to drill swelling or sloughing shales, salt, gypsum, anhydrite or other evaporite formations, hydrogen sulfide-containing formations, and hot (greater than 300 degrees Fahrenheit) holes, but may be used in other holes penetrating a subterranean formation as well.

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Gravel packing is becoming an increasingly common or favored method of completing a wellbore (as opposed to cementing and perforating a well). Well service fluids are used to carry the gravel into the well and place it at the gravel packing site. As used herein, the term "gravel," shall be understood to include not only natural gravel but other proppant type materials, natural and man-made or synthetic, such as, for example, sand, pebbles, and synthetic beads. Generally, the well's bottom hole pressure, which must be equalized, determines the minimal density of the fluid needed. A solids free water or brine, typically called "clear brine," has been commonly used to carry gravel for gravel packing a well and for other well servicing operations. Such fluid typically contains a zinc or calcium or sodium halide, such as sodium chloride, dissolved therein to provide the desired density to the fluid.

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However, when a well has been drilled with an oil or synthetic-based drilling fluid, "clear brine" fluids can be incompatible with the drilling fluid or with the subterranean formation having characteristics that necessitated the use of the oil or synthetic-based drilling fluid such as a brine -sensitive formation. Clean hydrocarbon oils would likely provide the most compatible or least damaging completion fluids, but such oils do not have the required densities and do not readily dissolve compounds that could provide the required densities.

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A need exists for an oil or synthetic-based, solids-free, well service fluid that is compatible with oil or synthetic-based drilling fluids and brine-sensitive formations and that

has the ability to be weighted to equal or greater than the bottomhole pressure requirements.

SUMMARY OF THE INVENTION

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A well service fluid has been discovered that can be made compatible with oil or synthetic-based drilling fluids and brine sensitive formations. This fluid comprises a solidsfree, invert emulsion comprising heavy brine, synthetic or oil, and an emulsifier sufficient for facilitating formation of an invert emulsion. The brine forms the internal phase of the emulsion and the synthetic forms the external phase of the emulsion.

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Preferably, synthetic is selected that is compatible with the oil or synthetic drilling fluid used to drill the wellbore to be serviced. The brine is made heavy with halide salts, preferably calcium bromide or zinc bromide or mixtures thereof, or cesium formate. Such halides confer density to the emulsion fluid without need for addition of other materials or solids.

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In the method of the invention, the emulsion of this invention for servicing a wellbore is preferably added directly to the wellbore, without dilution by or addition to other fluids. An example of a well service or treatment particularly suitable for the invention is gravel packing. Other suitable treatments include workover operations and use of the emulsions as packer fluids and displacement fluids.

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The invention has particular utility in horizontal wells.

DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS

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The present invention provides an improved well treatment method and composition employing water-in-oil emulsions, also called invert emulsions. These emulsions of the invention are especially useful for gravel packing and workover operations. The emulsions of the invention may also be useful as a packer fluid, a production fluid, a displacement fluid, and a drilling fluid, especially for drilling through the pay zone of a subterranean hydrocarbon-bearing formation. It is believed that the emulsions of the invention may further have utility in a single fluid system for drilling and completing wells, especially for construction and production of horizontal wells and wells penetrating brine-sensitive formations.

The internal phase of the emulsion of the invention comprises a high density brine ("heavy brine"), made heavy or dense with halide salts, such as, for example, calcium

bromide or zinc bromide. The external phase of the emulsion comprises synthetic or oil that is preferably compatible with oil or synthetic-based drilling fluids. An emulsifier blended with the brine and the oil facilitates formation of the emulsion. The brine portion of the emulsion dissolves salts to enhance and control the density of the fluid, while the oil portion of the emulsion allows for compatibility with oil-based drilling fluids and with brinesensitive formations.

Most preferably, filtered brine or "clear brine" is used for the emulsion. Preferred oils or synthetics for the emulsion are synthetic oils such as olefins and n-alkanes and some crude oils (preferably lighter crude oils) and distilled hydrocarbons. Preferred emulsifiers comprise polyolesin amides and alkeneamides. Preferably, the emulsifier will be relatively passive, stable at temperatures as high as 400 degrees Fahrenheit, and suitable for use with a wide variety of oils, synthetics and produced crudes (or crude oils). Also preferably, the emulsifer will be useful in high density brines. An example of such a preferred emulsifier is "BromiMulTM", an oil wetting nonionic surfactant emulsifier available from Baroid, a Halliburton company, in Houston, Texas. Preferred densities of salts in the emulsions may be determined or controlled by the quantity of halide salt added to the brine and the particular halide chosen. Generally, the more salt added, the greater the density, and the heavier the salt added (depending on its composition), the greater the density. For example, preferably about 10 to 15 or more pounds per gallon of calcium bromide or zinc bromide may be used for the invention, while up to or about 20 or more pounds per gallon of cesium formate may alternatively be used. Various quantities of oil or synthetic and water or brine may be used. so long as an invert (water-in-oil) emulsion is created and the rheology of the fluid is suitable for the intended use. For gravel packing, a 50:50 ratio of brine and oil is preferred, having a density preferably in the range of about 9 to about 13 pounds per gallon. The quantity of emulsifier used is an amount sufficient to facilitate formation of the emulsion. Generally, about 5 to about 20 pounds per barrel is sufficient. An advantage of the invention is that the emulsion provides fluid density without need for the addition of solids commonly used in the industry to add density to well service fluids and drilling fluids to satisfy bottomhole pressure requirements.

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After preparation, the invert emulsions are preferably used directly for the well service treatment. For example, for gravel packing, the gravel is carried by the emulsions comprising the well service fluid into the wellbore site for gravel packing. The emulsions will, however, contact drilling mud or drilling fluid used in the well and the oil or synthetic comprising the emulsion should preferably be compatible with the oil or synthetic in the drilling mud. Best

results are expected to be obtained when the emulsions are used in gravel packing where the gravel is being continuously deposited during the packing operation.

Experiments

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Samples of water-in-oil invert emulsions were prepared using a solids free or filtered brine, and PETROFREE LETM synthetic base oil, obtained from Baroid Technology, Inc., a Halliburton Company, Houston, Texas, in oil to water ratios ("OWR") of 50:50, 40:60, and 30:70, and a Bromi-MulTM emulsifier, obtained from Halliburton Energy Services, Inc., Houston, Texas. Calcium bromide (14.2 pounds per gallon) or calcium/zinc bromide (19.2 pounds per gallon) was added to the samples to increase the densities of the fluids. The term "calcium/zinc bromide" as used herein shall be understood to mean a mixture of calcium bromide and zinc bromide.

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All samples of the emulsions were multi-mixed for 30 minutes and rolled overnight at 150 degrees Fahrenheit before testing. Rheology was measured at 120 degrees Fahrenheit.

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As shown in Table 1, the 50:50 OWR fluids produced emulsions with densities of 10.2 pounds per gallon using calcium bromide and 12.2 pounds per gallon using calcium/zinc bromide. As shown in Table 2, the 40:60 OWR fluids produced emulsions with densities of 11.0 pounds per gallon using calcium bromide and 13.7 pounds per gallon using calcium/zinc bromide. As shown in Table 3, the 30:70 OWR fluids produced emulsions with densities of 11.6 pounds per gallon with calcium bromide and 14.7 pounds per gallon with calcium/zinc bromide. The viscosity of the 30:70 OWR fluids was too high for the intended use of gravel packing but the viscosities of the 40:60 OWR fluids and the 50:50 OWR fluids were suitable.

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All samples were static aged at 300 degrees Fahrenheit for 16 hours and all exhibited some top-oil separation--the 50:50 OWR fluid exhibited the most--but the emulsions did not break. When stirred, the emulsions remained stable at ambient temperature.

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In testing with sand and gravel for use in gravel packing horizontal wells, the emulsions were found to be particularly suitable for gravel packing operations where continuous deposition of proppant grains is expected and needed. Favorably, the emulsions tested were not successful with static sand suspension (i.e., the emulsions did not suspend sand in static conditions). These test results are desired for emulsions intended for use in horizontal or directional wells, where gravity does not facilitate deposition of suspended gravel and thus suspended gravel is undesirable.

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TABLE 1 (50/50 OWR Samples)

Sample No.	1	2	3	4	5	
PETROFREE LE™, bbl	0.5	0.5	0.5	0.5	0.5	0.5
14.2 lb/gal CaBr ₂ , bbl	0.5	0.5	0.5			
19.2 lb/gal CaBr ₂ /ZnBr ₂ , bbl				0.5	0.5	0.5
BROMI-MUL, lb	5	10	15	5	10	15
Rollsampies	150°F	16hrs.		Testat	120°F	
600 rpm	'34	41	42	26	30	36
300 rpm	17	21	22	13	14	18
200 грт	11	14	14	9	10	12.5
100 rpm	6	7	7	5	5	6.5
6 rpm	0.5	0.5	0.5	0.5	0.5	1
3 rpm	0.5	0.5	0.5	0.5	0.5	0.5
Plastic viscosity, cP	17	20	20	13	14	18
Yield point, lb/100ft ²	0	1	2	0	0	0
10 sec gel, lb/100ft ²	0	0	0	0	0	0
10 min gel, lb/100ft ²	0	0	0	0	0	0
Electrical stability, volts	160	168	170	65	80	85
Static aged 300°F	lehrs.	Multi	- mixed	10 min	Testat	12(
600 rpm	34	40	42	33	33	37
300 rpm	17	20	21	17	17	19
200 rpm	12	14	15	12	12	13
100 rpm	6	7	7	6	6	7
6 грт	0.5	1	1	1	0.5	1
3 rpm	0.5	0.5	0.5	0.5	0.5	0.5
Plastic viscosity, cP	17	20	21	16	16	18
Yield point, lb/100ft ²	0	0	0	1	1	1
10 sec gcl, lb/100ft ²	0	0	0	0	0	0
10 min gel, lb/100ft ²	0	0	0	0	0	0
Measured density, lb/gal		10.2			12.2	

TABLE 2
(40/60 OWR Samples)

	Sample No.	1	2	3	4
	PETROFREE LE™, bb1	0.4	0.4	0.4	0.4
5	14.2 lb/gal CaBr ₂ , bbl	0.6	0.6		
	19.2 lb/gal CaBr ₂ /ZnBr ₂ , bbl			0.6	0.6
	BROMI-MUL, Ib	10	15	10	15
	Rollsamples 150°F 16 hrs.,	Test	a t	1200	F
	600 rpm	100	133	76	96
10	300 rpm	53	71	40	50
	200 rpm	36	48	27	35
	100 rpm	19	26	14	16
	6 rpm	1.5	2	1	1.5
	3 rpm	1	1	0.5	1
15	Plastic viscosity, cP	47	52	36	46
	Yield point, lb/100ft ²	6	9	4	4
	10 sec gel, lb/100ft ²	1	1	. 1	1
	10 min gel, lb/100ft ²	1	1	1	1
	Electrical stability, volts	250	295	90	100
20	Static aged 300°F 16hrs, Multi-mixed	10 m i n,	Test	a t	120°F
	600 rpm	117	133	74	96
	300 rpm	62	72	39	45
	200 rpm	42	48	26	31
	100 rpm	22	25	14	16
25	6 rpm	2	2	1.5	1.5
	3 rpm	1	1	1	1
	Plastic viscosity, cP	55	62	35	41
	Yield point, lb/100ft ²	7	9	4	4
	10 sec gel, lb/100ft ²	1	1	1	1
30	10 min gel, lb/100ft ²	2	2	1	1
	Measured density, lb/gal		11.0		13.7

TABLE 3
(30/70 OWR Samples)

	Sample N .	1	2	3	4
	PETROFREE LE™, bbl	0.3	0.3	0.3	0.3
5	14.2 lb/gal CaBr ₂ , bbl	0.7	0.7		
	19.2 lb/gal CaBr ₂ /ZnBr ₂ , bbl			0.7	0.7
ĺ	BROMI-MUL, lb	10	15	10	15
	Roll samples 150°F 16 hrs.,	Test	a t	1200	F
	600 rpm	Off scale	Off scale	Off scale	Off scale
10	300 rpm	Off scale	Off scale	195	280
	200 rpm	278	Offscale	147	210
. [100 rpm	186	204	90	129
	6 rpm	39	41	125	17
	3 rpm	27	28	8	10
15	Plastic viscosity, cP				
_	Yield point, lb/100ft ²			******	
	10 sec gel, lb/100ft ²	27	28	7	10
_	10 min gel, lb/100ft ²	29	30	8	11
·	Electrical stability, volts	180	200	90	100
20	Static aged 300°F 16hrs, Multi-mixed	10 min,	Test	a t	120°F
	600 rpm	Off scale	Off scale	Off scale	Off scale
	300 rpm	Off scale	Off scale	244	299
•	200 rpm	Off scale	Off scale	189	229
	100 rpm	230	230	121	147
25	6 rpm	59	55	22	26
	3 rpm	44	41	14	18
	Plastic viscosity, cP				
	Yield point, lb/100ft ²				
	10 sec gel, lb/100ft ²	44	41	14	18
30	10 min gel, lb/100ft ²	47	42	15	19
	Measured density, lb/gal	11.6		14.7	

Samples of the emulsions of this invention were found to be compatible with a commonly used oil or syn hetic-based drilling fluid, COREDRIL-NTM, obtained from Halliburton Energy Services. Inc., Houston, Texas. An 11.0 lb/gal. COREDRIL-N fluid was prepared with the formulation shown in Table 4, and placed in an API filter press.

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TABLE 4

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PETROFREE LE™, bbl	0.7900
EZ-CORE™, lb	3
Lime, lb	3
Bentone 34, lb	8
DURATONE® HT, lb	5
BARABLOK™, lb	5
BARACARB® 5, lb	35
Barite, lb	182

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Three milliliters of filtrate were collected at 100 psi over 30 minutes. The cell was then opened and the COREDRIL-N fluid was poured out into a jar leaving the filter cake at the bottom of the cell. A sample of a 40/60 OWR invert emulsion of this invention comprising calcium bromide and 15 pounds of BROMI-MULTM fluid was poured into the cell on top of the COREDRIL-NTM filter cake. The cell was repressured and filtrate collected overnight. After 16 hours approximately 5 cc of filtrate had been collected. The collected filtrate was dark brown in color and normal in appearance. This filtrate was mixed with the filtrate previously collected from the COREDRIL-NTM sample. The two filtrates were compatible. A small amount of the emulsion was mixed with the combined filtrates and was again compatible.

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This compatibility test was repeated, substituting for the emulsion used in the previous test a 40/60 OWR invert emulsion of this invention comprising calcium/zinc bromide and BROMI-MUL.TM Tests results were similar, indicating no incompatibility.

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Samples of emulsions of this invention were tested for filtration ability and passed satisfactorily. Two 40/60 OWR invert emulsions were prepared with BROMI-MULTM, one

comprising 14.2 lb/gal. calcium bromide and the other comprising 19.2 lb/gal. calcium/zinc bromide, as shown in formulation Table 5.

TABLE 5

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PETROFREE LE™, bbl	0.4	0.4
14.2 lb/gal CaBr ₂ , bbl	0.6	
19.2 lb/gal CaBr ₂ /ZnBr ₂ , bbl		0.6
BROMI-MUL™, lb	10	10
Filtration time, 350 ml, minutes	6.5	2.5

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Each sample was placed in an API filtration cell containing 1.6 micron glass filter fiber paper. Filtration was continued until all of the sample passed through the paper. The filtered sample containing calcium bromide showed good stability with no breakdown of the emulsion. The sample containing calcium/zinc bromide showed some emulsion breakdown 24 hours after filtration.

Samples of emulsions of this invention were tested for toxicity and passed satisfactorily.

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Tests were also conducted to ascertain the effect if any of the invert emulsions of the invention on the subterranean formation. That is, the emulsion was tested for filter cake erosion and return permeability. These tests were conducted with core floods in a laboratory. The invert emulsion was comprised of ELF-GIRASSOLTM (available from Elf Aquitaine in France), which is a diesel oil, heavy brine, and BROMI-MULTM emulsifier. A filter cake was deposited on the core under pressure. The core was an inert, uniform ceramic material to simulate formation material. The emulsion was circulated in the core as in a gravel pack situation and did not erode the cake to the point of lost circulation. Also, the return permeability with the emulsion was greater than the return permeability with the drilling fluid used to build the filter cake. These tests showed the emulsion could be used for gravel packing without causing formation damage.

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Further tests were conducted to simulate a field treatment, particularly gravel packing, using invert emulsions according to the present invention. These tests employed a 12 foot acrylic cylinder with a wire wrapped screen inside. A base line water pack preceded the test

with a 9.4 pounds per gallon (ppg) invert emulsion of this invention as a gravel pack fluid. The emulsion comprised 181 gallons HDF-2000[™] (a mineral oil available from Total of France), 105 gallons calcium bromide solids-free brine (having 14.2 pounds per gallon calcium bromide), and 14 gallons BROMI-MUL[™] emulsifier. In the test, the gravel placement with the emulsion was steady and even, comparable to and even better than the gravel placement with water or brine as commonly used for gravel packing.

This emulsion further tested successfully for filterability in a diatomaceous earth filter press. Fluid samples were taken after each pass of the emulsion through the filter press. The emulsion maintained stability and was filterable even after several passes through the filter. Further, the emulsion did not damage the permeability of the 20/40-mesh sand pack used in the test. Analysis of the emulsion samples caught after each pass through the filter press showed that the samples maintained their rheological properties. After the samples were allowed to sit undisturbed (static) for over 24 hours, the emulsion samples showed little if any separation of the synthetic and brine phases, further indicating their stability and success in this test.

The foregoing description of the invention is intended to be a description of a preferred embodiment. Various changes in the details of the described composition and method can be made without departing from the intended scope of this invention as defined by the appended claims.

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1	We c	We claim:					
1	1.	. A method for treating a subterranean brine-sensitive formation surrounding a					
2	1.	wellbore, which comprises:					
3		contacting the formation with a solids-free, invert emulsion comprising:					
4	,	heavy brine as the internal phase of said emulsion, an oil or					
5		synthetic as the external phase of said emulsion.					
1	2.	The method of claim 1 further comprising an emulsifier in sufficient quantity to					
2		facilitate formation of the emulsion.					
1	3.	The method of claim 1 where said treatment is gravel packing.					
1	4.	The method of claim 1 where said treatment is a workover operation.					
1	5.	The method of claim 1 where said treatment employs said emulsion as a displacement					
2		fluid or as a packer fluid.					
1	6.	The method of claim 1 where said treatment is drilling through a pay zone of said					
2		formation.					
1	7.	The method of claim 1 where said emulsion comprises salts or oils sufficient to give					
2		the emulsion a specific density suited for the particular treatment.					
1	8.	The method of claim 1 wherein said wellbore is at least partially horizontal.					
1	9.	The method of claim 2 wherein said heavy brine comprises calcium bromide or zinc					
2		bromide.					
1	10.	The method of claim 2 wherein said emulsifier is a non-ionic surfactant.					

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about 400 degrees Fahrenheit.

The method of claim 10 wherein said emulsifier is stable at temperatures as high as

1	12.	A method for grave! packing a wellbore penetrating a subterranean formation
2	•	comprising:
3		preparing an invert entulsion comprising oil or synthec, clear brine, and halide salts;
4	•	injecting said emulsion with gravel into said wellbore such that said emulsion is a
5		carrier for said gravel; and
6		depositing said gravel on the sides of said wellbore.
1	13.	The method of claim 12 wherein said halide salts are selected from the group
2		consisting of calcium bromide, zinc bromide, cesium formate, and mixtures of
3		calcium bromide and zinc bromide.
1	14.	The method of claim 12 wherein said halide salts are added in quantities sufficient to
2		give the emulsion a density suitable for the intended purpose of the emulsion.
1	15.	The method of claim 12 wherein said wellbore is horizontal.
1	16.	The method of claim 15 wherein said wellbore was drilled using an oil-based or
2		synthetic-based drilling fluid and said emulsion is compatible with said drilling fluid.
1	17.	A well service fluid comprising a solids-free, invert emulsion comprising clear heavy
2		brine as the internal phase of said emulsion, synthetic as the external phase of said
3		emulsion, and an emulsifier to facilitate formation of said emulsion.
1	18.	The well service fluid of claim 17 wherein said emulsifier is comprised of a non-ionic
2		surfactant.
1	19.	A fluid for gravel packing a wellbore in a subterranean formation drilled with an oil-
2		based or synthetic-based drilling mud, said fluid comprising:
3		an oil or synthetic compatible with said drilling mud;
4		a solids-free, heavy brine containing halide salts; and
5		an emulsifier in sufficient quantity to facilitate formation of an invert emulsion with
6		said compatible oil or synthetic and said heavy brine.

1	20.	The fluid of claim 19 wherein said halide salts are selected from the group consisting
2 '	•	of calcium bromide, zinc bromide, cesium formate, and mixtures of calcium bromide
3		and zinc bromide, and are present in sufficient quantity to lend sufficient density to
		said emulsion to enable said emulsion to carry gravel.

INTERNATIONAL SEARCH REPORT

CT/US 01/01973

CLASSIFICATION OF SUBJECT MATTER C09K7/06 E21B43/22 E21B43/04 E21B43/02 According to International Patent Classification (IPC) or to both national classification and IPC B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) E21B C09K Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Electronic data base consulted during the international search (name of data base and, where practical, search terms used) EPO-Internal C. DOCUMENTS CONSIDERED TO BE RELEVANT Calegory 9 Citation of document, with indication, where appropriate, of the relevant passages Relevant to claim No. X EP 0 561 607 A (LUBRIZOL CORP) 1,2,7,9, 22 September 1993 (1993-09-22) 10,17 page 2, line 51 -page 3, line 5 page 15, line 49 -page 16, line 16 Ρ, χ WO 00 56835 A (MI LLC) 1,2,4,6, 28 September 2000 (2000-09-28) 7,9,10, page 1, line 1 - line 14 page 3, line 24 - line 28 page 4, line 5 - line 9 page 5, line 10 - line 14 page 6, line 22 -page 7, line 15 page 8, line 3 -page 9, line 24 χ 1,3,7,12 US 3 675 717 A (GOINS WILLIAM C JR ET AL) 11 July 1972 (1972-07-11) column 2, line 34 -column 3, line 15 Further documents are listed in the continuation of box C. X Patent family members are tisted in annex. Special categories of cited documents: "T" later document published after the international filing date or priority date and not in conflict with the application but "A" document defining the general state of the art which is not cited to understand the principle or theory underlying the considered to be of particular relevance "E" earlier document but published on or after the international "X" document of particular relevance; the claimed invention filing date cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another *Y* document of particular relevance; the claimed invention citation or other special reason (as specified) cannot be considered to involve an inventive step when the *O* document referring to an oral disclosure, use, exhibition or document is combined with one or more other such docu ments, such combination being obvious to a person skilled other means document published prior to the international filing date but later than the priority date claimed "&" document member of the same patent family Date of the actual completion of the international search Cate of mailing of the international search report 24/07/2001 13 July 2001 Name and mailing address of the ISA Authorized officer European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk

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INTERNATIONAL SEARCH REPORT

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